

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Art Unit : 1764
Examiner : Thuan D. Dang
Serial No. : 09/589,588
Filed : June 8, 2000
Inventors : Akira Kitamura
: Ryoji Ichioka
: Shinobu Yamakawa
Title : METHOD FOR CONVERTING
: AROMATIC HYDROCARBONS

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Customer No.: 035811

Docket No.: 1197-00

Confirmation No.: 1857

Dated: September 12, 2003

Commissioner for Patents
P.O. Box 1450
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Sir:

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English Translation of Priority Document JP 11-169100

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Date: 12 SEP 2003

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LETTER

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Sir:

We submit herewith an English translation of Japanese Patent Application No. 11-169100,
filed June 16, 1999, the certified copy of which was filed on June 8, 2000 with the Application.

Respectfully submitted,



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[List of documents submitted]

[Name of the document] Specification 1

[Name of the document] Abstract 1

[Necessity of proof] Required

[Title of the document] Specification

[Title of the invention] Method for converting aromatic hydrocarbons

[Claims]

[Claim 1] A method for converting aromatic hydrocarbons, comprising the step of bringing aromatic hydrocarbons containing benzene and 1 wt% or less of non-aromatic compounds into contact with a catalyst.

[Claim 2] A method for converting aromatic hydrocarbons, according to claim 1, wherein the non-aromatic compounds are removed from the aromatic hydrocarbons containing benzene, to achieve a non-aromatic compound content of 1 wt% or less, and the remaining aromatic hydrocarbons are brought into contact with a catalyst.

[Claim 3] A method for converting aromatic hydrocarbons, according to claim 1 or 2, wherein the conversion of the aromatic hydrocarbons is a transalkylation reaction.

[Claim 4] A method for converting aromatic hydrocarbons, according to any one of claims 1 through 3, wherein the raw material contains alkyl aromatic hydrocarbons with 9 and more carbon atoms.

[Claim 5] A method for converting aromatic hydrocarbons, according to claim 4, wherein the benzene and aromatic hydrocarbons with 9 and more carbon atoms in the raw material are decreased, while the aromatic hydrocarbons with 7 and 8 carbon atoms in the product are increased.

[Claim 6] A method for converting aromatic hydrocarbons, according to any one of claims 1 through 5, wherein the catalyst contains a zeolite.

[Claim 7] A method for converting aromatic hydrocarbons, according to any one of claims 1 through 6, wherein the catalyst contains at least one selected from the metals of group VIB, group VIIB and group VIII of the periodic table.

[Claim 8] A method for converting aromatic hydrocarbons, according to any one of claims 1 through 7, wherein the catalyst contains mordenite and rhenium.

[Detailed description of the invention]

[0001]

[Technical field to which the invention pertains]

The present invention relates to a method for converting aromatic hydrocarbons. In more detail, it relates to an aromatic compound-converting method for converting a raw material consisting of aromatic hydrocarbons containing benzene and 1 wt% or less of non-aromatic compounds.

[0002]

[Prior art]

Recent requirements and regulations for environmental issues are increasingly severe. Especially for gasoline, the influence of the benzene contained in gasoline on the human health becomes a serious problem, and in USA, a regulation is already imposed on the benzene content of gasoline. Also in Japan and Europe, there are movements for regulations, and oil companies are making efforts to decrease the benzene of gasoline.

[0003]

To meet the demand for high-octane fuels in the market, benzene and other alkyl aromatic hydrocarbons such as toluene and xylene, respectively high in octane value, are contained in the conventional gasoline at high concentrations as important base materials for gasoline. Gasoline is usually produced in refining processes such as catalytic reforming and cracking, and the distillates of respective processes contain benzene. As for the removal of benzene from gasoline for the sake of environment, since the benzene contained in the reformed gasoline only produced in Japan amounts to about 700,000 tons, the effective use of benzene is an urgent issue. As a method for using benzene, known is a technique, in which the transalkylation reaction between benzene and aromatic hydrocarbons with 9 and more carbon atoms is used for producing toluene and xylene. Toluene and xylene are said to have no problem for the time being in view of the influence on the human health unlike benzene, and furthermore since they are higher in octane value than benzene, the reaction is preferred also in view of enhancing the octane value of gasoline.

[0004]

Usually the benzene to be removed from gasoline is separated by means of distillation. The benzene fraction contains a large amount of non-aromatic compounds close to benzene in boiling point, above all, non-aromatic hydrocarbons such as paraffins, olefins and naphthenes. If the transalkylation reaction between the benzene fraction containing a large amount of non-aromatic compounds and aromatic hydrocarbons with 9 and more carbon atoms is carried out in the presence of hydrogen, the reaction for cracking paraffins and the like occurs concurrently with the transalkylation of aromatic compounds. If the cracking reaction as a side reaction occurs, hydrogen consumption increases to pose a problem that the heat generation by cracking becomes large.

[0005]

US Patent 5347061 discloses a method, in which the benzene in the gasoline fraction and the hydrocarbons with 9 and more carbon atoms are converted into alkyl aromatic compounds with 7 and 8 carbon atoms. It relates to a process, in which a stream comprising benzene-rich hydrocarbons with 6 carbon atoms and a stream comprising hydrocarbons with 9 and more carbon atoms respectively obtained by distilling reformed gasoline are subjected to a cracking reaction, transalkylation reaction and alkylation reaction in the presence of an acidic metallocsilicate catalyst for producing aromatic hydrocarbons with 7 and 8 carbon atoms. However, in the US Patent 5347061, since hydrocarbons with 9 and more carbon atoms are positively catalytically cracked for use as an alkylating agent for benzene alkylation, the presence of non-aromatic hydrocarbons is essentially required.

[0006]

JP9-38497A discloses an aromatic hydrocarbon-converting method, in which a benzene-containing fraction obtained by fractional distillation of the product obtained by catalytically reforming naphtha and a trialkylbenzene-containing fraction obtained by fractional distillation of the product obtained by catalytic cracking, are used as raw materials for a transalkylation reaction in the presence of a crystalline aluminosilicate catalyst

loaded with a metal of group VIII of the periodic table, to produce a reaction product mainly containing monoalkylbenzenes and dialkylbenzenes. For the crystalline aluminosilicate, it is described that mordenite with a low shape selectivity index is low in practical value since it is greatly deteriorated by caulking, and that TSZ is preferred in view of overall adsorption capacity and shape selectivity index.

[0007]

JP9-155198A discloses a method, in which a catalyst obtained by letting a carrier containing a zeolite with a maximum micropore size of 0.6 to 1.0 nm and a $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio of 50 or more be loaded with a metal selected from the metals of groups VIII and VIA of the periodic table or any of its compounds, is used to convert aromatic hydrocarbon compounds with 9 and more carbon atoms contained in a feedstock having a boiling point in a specific range and not containing benzene, into toluene and aromatic hydrocarbon compounds with 8 carbon atoms in the presence of hydrogen. It is also described that mordenite is preferred as the zeolite and that nickel, palladium or molybdenum is preferred as the metal.

[0008]

JP9-38505A discloses a method, in which a catalyst obtained by letting a carrier containing a zeolite with a maximum micropore size of 0.6 to 1.0 nm and a $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio of 50 or more be loaded with a metal selected from the metals of groups VIII and VIA of the periodic table or any of its compounds, is used to convert the benzene and aromatic hydrocarbon compounds with 9 and more carbon atoms in a feedstock having a boiling point in a specific range, into toluene and aromatic hydrocarbon compounds with 8 carbon atoms in the presence of hydrogen.

[0009]

JP9-187658A discloses a method, in which a catalyst obtained by letting a carrier containing a zeolite with a maximum micropore size of 0.6 to 1.0 nm and a $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio of less than 50 be loaded with a metal selected from the metals of groups VIII and VIA of the periodic table or any of its compounds, is used to convert the

benzene and aromatic hydrocarbon compounds with 9 and more carbon atoms in a feedstock having a boiling point in a specific range, into toluene and aromatic hydrocarbon compounds with 8 carbon atoms in the presence of hydrogen.

[0010]

WO98-12159 discloses a process, in which an aromatic raw material containing at least 20% or more of aromatic compounds with 9 carbon atoms having an ethyl group or propyl group is brought into contact with a catalyst containing mordenite loaded with palladium and getting rid of aluminum to have a silica/alumina ratio of 12 to 30 in the presence of hydrogen, for conversion, to obtain a toluene- and xylene-rich product.

[0011]

WO96-24568 discloses a process, in which a raw material containing aromatic hydrocarbons with 9 and more carbon atoms, benzene and/or toluene is brought into contact with a catalyst containing a zeolite with a control index of 0.5 to 3, having a hydrogenation region and treated to inhibit nuclear hydrogenation capability, for conversion into aromatic compounds with 9 and less carbon atoms.

[0012]

US Patent 5406016 discloses a process, in which a 12-membered ring zeolite is used for converting benzene and alkyl aromatic compounds with 10 and more carbon atoms and having two or more alkyl groups, into methylbenzenes under specific reaction conditions.

[0013]

However, the above-mentioned techniques are not sufficient in the efficiency at which aromatic compounds are converted, and the documents do not describe the method for dealing with non-aromatic compounds at all.

[0014]

[Problem to be solved by the invention]

The object of this invention is to provide a method for converting a raw material consisting of aromatic hydrocarbons containing benzene, which consumes less hydrogen, and little

deteriorates the catalyst, allowing more effective use of benzene.
[0015]

[Means for solving the problem]

The inventors studied intensively to solve the above-mentioned problem, and as a result, found that if the non-aromatic compounds, above all, non-aromatic hydrocarbons such as paraffins, olefins and naphthenes contained in a raw material consisting of aromatic hydrocarbons are decreased, both the hydrogen consumption and the deterioration of the catalyst can be decreased, when the aromatic hydrocarbons are converted. As a result, the heat generation caused by the cracking of non-aromatic compounds in the reaction can also be decreased.

[0016]

This invention provides an aromatic hydrocarbon-converting method for converting a raw material consisting of aromatic hydrocarbons containing benzene and 1% or less of non-aromatic compounds.

[0017]

This invention is a method for converting aromatic hydrocarbons, comprising the step of bringing aromatic hydrocarbons containing benzene and 1 wt% or less of non-aromatic compounds, into contact with a catalyst.

[0018]

[Modes for carrying out the invention]

This invention is described below in more detail.

[0019]

This invention converts a raw material consisting of aromatic hydrocarbons containing benzene and 1 wt% or less of non-aromatic compounds. It is preferred that the raw material used has gotten rid of non-aromatic compounds. The aromatic hydrocarbons obtained by conversion can also be used as an ordinary chemical raw material, or as a base material of gasoline short of removed benzene, and the benzene removed from gasoline can be effectively used.

[0020]

The non-aromatic compounds in this invention refer to the

compounds other than aromatic hydrocarbons, and particularly refer to non-aromatic hydrocarbons such as paraffins, olefins and naphthenes. This invention is characterized in that the amount of the non-aromatic compounds is 1 wt% or less based on the weight of the benzene-containing aromatic hydrocarbons used as the raw material.

[0021]

The conversion of aromatic hydrocarbons in this invention particularly refers to at least one of transalkylation reaction, dealkylation reaction and disproportionation reaction. The reaction is carried out under the conditions, in which at least one of transalkylation reaction, dealkylation reaction and disproportionation reaction takes place, usually in the presence of hydrogen. The reaction pressure is in a range of 0.1 to 100 MPa. A preferred range is 0.5 to 60 MPa, and a more preferred range is 1 to 50 MPa. If the pressure is too low, the catalyst is deteriorated quickly, and the reaction rate also becomes low. If the pressure is too high, an economic disadvantage in view of equipment is inevitable. The reaction temperature is in a range of 200 to 650°C, and a preferred range is 250 to 500°C. If the reaction temperature is too low, the reaction rate becomes low. If it is too high, cracking occurs heavily and the catalyst is deteriorated quickly. The hydrogen flow rate as hydrogen/raw material (mol/mol) is in a range of 0.1 to 20. A preferred range is 0.5 to 10. If the flow rate is too low, the catalyst is deteriorated quickly, and if it is too high, an economical disadvantage is inevitable.

[0022]

The raw material used in this invention consists of aromatic hydrocarbons containing benzene. The aromatic hydrocarbons may include benzene, toluene, xylene, ethylbenzene, trimethylbenzene, ethyltoluene, propylbenzene, tetramethylbenzene, ethylxylene, diethylxylene, propyltoluene, and any other aromatic hydrocarbons. In the case where toluene and/or xylene is taken out as a product, it is necessary that the amount of toluene and/or xylene contained in the raw material is smaller than the corresponding content in

the equilibrium composition.

[0023]

As for the raw material, catalytically reformed oil or the like consisting of the fractions in the boiling point range of gasoline usually obtained from each refining process can be used as the raw material. Furthermore, feedstocks obtained from respective refining processes can also be used in combination as the raw material. A feedstock, especially for example the feedstock delivered from a reforming reactor is separated into fractions of adequate boiling point ranges such as C₅ fraction, C₆ fraction, C₇ fraction, C₈ fraction and C₉ + fraction. In the reaction of this invention, the separated fractions can be used in combination, or a feedstock can also be used without being separated. In any case, it is essentially required that the raw material contains benzene.

[0024]

In the reforming reaction, in general a fraction with less carbon atoms is higher in the non-aromatic compound content, and it can happen that a C₆ fraction becomes more than 50% in the non-aromatic compound content. It is preferred that these non-aromatic compounds are separated from aromatic compounds by means of distillation or extraction. The raw material that has gotten rid of non-aromatic compounds like this is then brought into contact with a catalyst, for conversion.

[0025]

The raw material consisting of benzene-containing aromatic compounds used in this invention is characterized in that the non-aromatic compound content is 1% or less. In the case where the non-aromatic compound content is more than 1 wt%, cracking consumes hydrogen heavily, and the heat generation due to cracking also becomes large, while the cracking product greatly deteriorates the catalyst. It is preferred that the non-aromatic compound content is 0.5 wt% or less. More preferred is 0.1 wt% or less.

[0026]

It is preferred that the non-aromatic compounds are removed

from the raw material by means of distillation or extraction. The non-aromatic compounds can be removed after the feedstock is separated into respective fractions by means of distillation or before distillation, if the removal is carried out before the conversion reaction. The removed non-aromatic compounds can be directly used as a base material of gasoline or can be used after they are enhanced in octane value by an isomerization process.

[0027]

The benzene content of the feedstock is not especially specified. However, for efficiently converting benzene into other aromatic hydrocarbons, the content should be 5 wt% to 80 wt%. A preferred range is 10 wt% to 70 wt%, and a more preferred range is 15 wt% to 60 wt%.

[0028]

Furthermore, in this invention, it is preferred that the feedstock contains alkyl aromatic hydrocarbons with 9 and more carbon atoms. Above all, it is preferred that alkyl aromatic hydrocarbons with 9 and 10 carbon atoms are contained. The alkyl aromatic hydrocarbons with 9 carbon atoms include trimethylbenzene, ethyltoluene and propylbenzene, and the alkyl aromatic hydrocarbons with 10 carbon atoms include tetramethylbenzene, ethylxylene, diethylxylene, propyltoluene and butylbenzene. Above all, trimethylbenzene and tetramethylbenzene are preferred. The amount of the alkyl aromatic hydrocarbons with 9 and more carbon atoms contained in the feedstock is not especially limited, but for efficient transalkylation with benzene, it is preferred that the amount is in a range of 5 wt% to 90 wt%. A more preferred range is 7 wt% to 85 wt%, and a further more preferred range is 10 wt% to 80 wt%.

[0029]

This invention is characterized in that, more particularly, a raw material consisting of aromatic hydrocarbons with 9 and more carbon atoms containing benzene and 1% or less of non-aromatic compounds is used to obtain a product by means of transalkylation reaction. The transalkylation reaction with the product can also

occur. The transalkylation reaction particularly includes the production of toluene and xylene from benzene and trimethylbenzene, the production of toluene, xylene and trimethylbenzene from benzene and tetramethylbenzene, the production of toluene from benzene and xylene, the production of xylene from toluene and trimethylbenzene, the production of xylene and trimethylbenzene from toluene and tetramethylbenzene, the production of ethylbenzene from benzene and diethylbenzene, and so on. Any other transalkylation reaction can also occur. These reactions decrease both benzene and aromatic hydrocarbons with 9 and more carbon atoms in the feedstock and increase the aromatic hydrocarbons with 7 and 8 carbon atoms in the product. It is preferred to remove the aromatic hydrocarbons with 7 carbons atoms and/or 8 carbon atoms from the product. The remaining product can also be recycled.

[0030]

In this invention, disproportionation reaction, dealkylation reaction or alkylation reaction can also occur in addition to transalkylation reaction.

[0031]

The catalyst used in this invention is a catalyst for converting aromatic compounds, particularly any catalyst that allows transalkylation to occur. Furthermore, disproportionation, dealkylation or alkylation can also occur. Preferred is a catalyst that can hold methyl groups and can selectively dealkylate ethyl groups and propyl groups, while having a concurrent transalkylation capability. Particularly a shape selective metallocsilicate catalyst is preferred, and above all a crystalline aluminosilicate is preferred. Especially a zeolite is preferred, and any zeolite such as mordenite, Y, X, beta or ZSM-5 can also be used. It is more preferred to use mordenite.

[0032]

The zeolite can also be ion-exchanged with adequate metal ions or hydrogen ions. A hydrogen zeolite is preferred.

[0033]

For the catalyst, as required, a binder can also be used. The

binder is not especially limited, and inorganic oxides such as alumina, silica alumina, titania and magnesia and clays such as montmorillonite, kaolin, sepiolite and acid clay can be used. Any one of them can be used as desired or two or more of them can also be used in combination. Alumina is preferred. The amount of the binder is not especially limited either, but it is preferred that the amount is 20 wt% to 60 wt% based on the weight of the catalyst. [0034]

It is preferred that the catalyst contains a region having hydrogenation activity for the purposes of enhancing the activity of the catalyst and elongating the life of the catalyst. Any catalyst having hydrogenation activity can be used as desired. The zeolite per se used as the catalyst may have the hydrogenation activity, but it is preferred that the catalyst contains at least one of the metals of groups VIB, VIIB and VIII of the periodic table. The metals of group VIB include, particularly, chromium, molybdenum and tungsten, and among them, molybdenum is preferred. The metals of group VIIB include, particularly, manganese, technetium and rhenium, and among them, rhenium is preferred. The metals of group VIII include iron, ruthenium, osmium, cobalt, rhodium, iridium, nickel, palladium and platinum, and among them, nickel, palladium and platinum are preferred. The most preferred metal is rhenium. It is preferred that any of these metals alone and/or any of compounds of the metals is loaded in the carrier. The method for loading a carrier with a metal is not especially limited, and any usually used method such as impregnation or CVD can be used. The amount of the metal loaded in the carrier is not especially limited either and can be selected as required, depending on reaction conditions, etc. It is preferred that the amount is in a range of 0.001 wt% to 10 wt% based on the weight of the catalyst. A more preferred range is 0.01 wt% to 5 wt%, and a further more preferred range is 0.02 wt% to 2 wt%.

[0035]

[Example]

This invention is described below more particularly in

reference to an example.

[0036]

Example 1

One hundred and five grams of powdery sodium type synthetic mordenite, 45 g of α -alumina, 12 g of alumina sol (containing 10 wt% of alumina), 10.5 g of alumina gel (containing 70 wt% of alumina) and an adequate amount of ion exchange water were kneaded for about 2 hours, and the mixture was molded into noodle-shaped pieces with an outer diameter of 1.2 mm and a length of 1.0 mm. The molded pieces were dried at 120°C for 16 hours, and 50 g of them (as dried at 120°C) were burned in air atmosphere at 400°C for 5 hours, and cooled. They were treated using 100 g of 10 wt% ammonia chloride aqueous solution at 80 to 85°C for 1 hour, and the liquid was filtered away, the residue being washed with water. Furthermore, the residue was treated using 100 g of 5 wt% tartaric acid aqueous solution at 80 to 85°C for 3 hours, and the liquid was filtered away, the residue being washed with water. The residue was immersed in 6.5 g of 5 wt% rhenium (VII) oxide aqueous solution at room temperature. It was dried at 120°C for 16 hours, and burned at 540°C for 8 hours in air atmosphere, to obtain an H type synthetic mordenite catalyst. The raw material composed as shown in Table 1 was distilled in a fixed bed flow reactor, for removing non-aromatic compounds, and the residue was brought into contact with the catalyst. The results are shown in Table 1. The reaction conditions were as follows.

[0037]

Reaction conditions:	Temperature	400°C
	Pressure	2.5 MPa ^{-G}
	Liquid space velocity	2.3 h ⁻¹
	Hydrogen/raw material	4.0 mol/mol

The hydrogen consumption during the reaction was 0.84 wt% based on the weight of the raw material, and the catalyst deterioration rate as measured in reference to the xylene concentration in the product was -0.04 wt%/100 hours.

[0038]

[Table 1]

Composition	Raw material before removal (wt%)	Raw material after removal (wt%)	Product (100 hours) (wt%)
Non-aromatic compounds	10.0	0.1	10.7
Benzene	21.3	22.6	8.0
Toluene	1.3	1.4	27.7
C ₈ aromatic compounds	0.3	0.3	33.5
C ₉ aromatic compounds	62.3	70.2	17.6
C ₁₀ aromatic compounds	4.8	5.4	2.5

Comparative Example 1

Under the conditions of Example 1, the raw material was brought into contact with the catalyst without being distilled. The results are shown in Table 2. The hydrogen consumption during the reaction was 1.14 wt% based on the weight of the raw material, and the catalyst deterioration rate was -0.08 wt%/100 hours.

[0039]

[Table 2]

Composition	Raw material (wt%)	Product (100 hours) (wt%)
Non-aromatic compounds	10.0	19.5
Benzene	21.3	7.7
Toluene	1.3	25.8
C ₈ aromatic compounds	0.3	29.7
C ₉ aromatic compounds	62.3	15.1
C ₁₀ aromatic compounds	4.8	2.2

[0040]

[Effects of the invention]

In this invention, a raw material consisting of aromatic hydrocarbons containing 1 wt% or less of non-aromatic compounds and further containing benzene is brought into contact with a catalyst for conversion into useful aromatic hydrocarbons such as toluene and xylene, while the hydrogen consumption and the decline of catalyst activity are kept small.

[Title of the document] Abstract

[Abstract]

[Problem to be solved] To provide a method for converting a raw material consisting of aromatic hydrocarbons containing benzene while keeping the hydrogen consumption and the catalyst deterioration small.

[Solution] To keep the amount of non-aromatic compounds in benzene-containing aromatic hydrocarbons at 1 wt% or less before bringing them into contact with a catalyst.

[Selected Drawing] Nil